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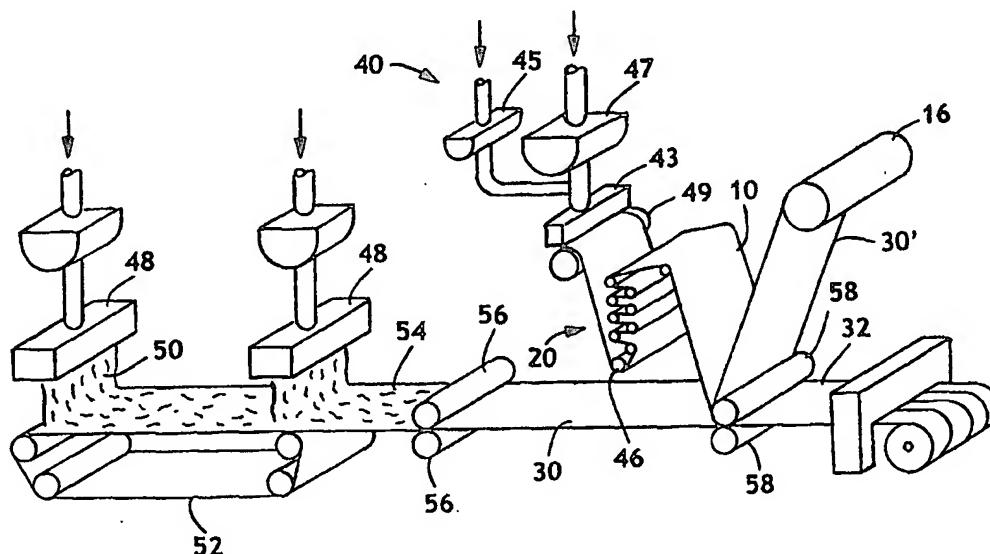




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(54) Title: PROCESS FOR MAKING A LAMINATE OF UNAGED FILM AND AN UNAGED NONWOVEN WEB AND PRODUCTS PRODUCED THEREFROM



## (57) Abstract

The present invention is directed to an in-line, continuous process for making a laminate of film and nonwoven web wherein the film and nonwoven web are made simultaneously, and are therefore nascent or unaged when directly formed into a laminate. In the process of the present invention, the film is formed in-line while the nonwoven web is formed, the newly formed film and nonwoven web being immediately laminated upon forming. Hence, the laminate is formed from "unaged" film and "unaged" nonwoven web. As used herein, the term "unaged", which is synonymous with "green", means that the film and nonwoven web are immediately laminated upon forming, for example, neither the film nor the nonwoven web is pre-wound into a roll prior to forming into a laminate.

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**PROCESS FOR MAKING A LAMINATE OF UNAGED FILM AND AN UNAGED  
NONWOVEN WEB AND PRODUCTS PRODUCED THEREFROM**

**Field of the Invention**

The present invention is directed to a process for making a laminate of unaged film and an unaged nonwoven web and products produced therefrom. More particularly, the present invention is directed to an in-line, continuous process for making the laminate wherein the film and nonwoven web are made simultaneously, then directly formed into a laminate.

**Background of the Invention**

Industry has long recognized the benefits of combining barrier properties of films and cloth-like attributes of nonwoven fabrics for various medical, personal care and commercial applications. Laminates have been traditionally produced using both aged film and an aged nonwoven web as well as by using aged film and an unaged nonwoven web, or vice versa, unaged film and an aged nonwoven web. As an example, a typical prior art process would entail first winding a pre-made film onto a roll and then later unwinding that roll of film while laminating to a nonwoven web as the nonwoven is being made, hence forming the laminate. There are many technical hurdles to making either a film or nonwoven web on a continuous basis. Furthermore, combining such complex processes as film making and nonwoven web making would place success at the mercy of simultaneous solution of technical issues of both systems. The difficulties inherent in each process, as proffered by Reliability Theory, would in fact lead one of ordinary skill in the art away from such a combination with no particular product attribute benefit expected, as will be discussed more fully below.

In addition, prior processes of unwinding either a preformed film or nonwoven web have had technical issues to overcome. One problem with the previously used unwind procedure relates to the additional steps required when one roll of aged film is depleted and a new roll of aged film must be substituted. Splicing is required to attach the end of the first roll of film to the beginning of the second roll of film. Since, in a continuous process, it is necessary to perform the splicing without stopping the machine, splicing is difficult since the rolls are usually rotating at high speeds. There are at least two known ways of performing the splicing. In one method, a festoon is

employed to take-up slack in the film which allows for zero speed splicing. The sheet of film is wrapped through the bars of the festoon which is expanded, for example, in a vertical fashion. When the splicing begins, the festoon is lowered, which allows the speed at the roll to approach zero. This method facilitates splicing in that movement of the rolls of aged film is eliminated. The disadvantage of such a process is that additional costly equipment (e.g. the festoon) must be purchased, maintained and operated.

Another method of splicing is generally known as "splicing on the fly," which requires splicing while the rolls are rotating. This method is conducted by slowing the rotational speed of the first roll, while speeding up the second roll, thereby matching surface speeds of the two rolls to perform the splice. The "flying splice" method can be a more cost effective method in that purchase, maintenance and operation of expensive festoon equipment is not required. As one of ordinary skill in the art is aware, however, it is much more difficult to "splice on the fly," especially when long rolls are employed and when speeds of between 400 and 1500 feet per minute (fpm) (or 122 to 457 meters per minute (m/min)) are employed.

Splicing may be achieved by many means as is known by those of ordinary skill in the art. One such method is to tape together the two film sheets. One disadvantage of taping is that the location of the taped portion must be monitored and later removed since the tape interferes with film properties such as breathability.

A further disadvantage of unwinding a pre-made, or aged, film, is that adjacent windings of the film tend to stick to each other (otherwise known as film blocking) resulting in tearing as the roll is being unwound from the roll. In fact, each film layer has an affinity for the next film layer and the layers prefer to adhere together rather than unwind. Both the nature of the material used to form the film and the compressive forces resulting from winding the film onto a roll tend to make the layers adhere together resulting in tears to the film when unwinding. Such tears result in lack of product uniformity, thereby decreasing the barrier properties. Additionally, the film may tear completely such that the process must be stopped requiring re-splicing of the film.

Also, it is not uncommon to use rolls of film that are as much as 120 inches (3.05 m) wide and weighing as much as 3000 lbs. (1360 kg). As one of ordinary skill in the art is aware, handling of such rolls is difficult and cumbersome to say the least.

The present invention avoids these and other difficulties by in-line manufacture of both the unaged nonwoven web and unaged film. Aside from overcoming the difficulties discussed above, there are advantages to utilizing the in-line, continuous

process of the present invention. One such advantage is that the unaged film has not had an opportunity to fully crystallize before laminating the film to the nonwoven. When a film has been wound onto a roll and allowed to age in storage for as little as 48 hours and as much as 2-3 weeks, the film temperature will cool and the film will crystallize. The unaged film of the present invention will remain more amorphous, meaning that it will be softer and easier to orient. Additionally, this amorphous characteristic may result in the ability to utilize a lower basis weight of film in either the pre-stretched film and/or the resulting laminate. In this way, the barrier and breathability properties of the film are maintained, or even possibly improved, while reducing the overall costs associated with manufacturing the film and resulting laminate.

All of these disadvantages of the prior known processes have been eliminated by the process of the present invention wherein unaged film is produced simultaneously with unaged nonwoven and immediately forming the laminate in an in-line, continuous process. Such laminates exhibit unexpected improvements in peel strength and hydrohead making them particularly useful in applications where the laminate will be converted into absorbent articles. The conversion process has been typically replete with problems associated with delamination. These problems have been improved when utilizing the laminates made by the process of the present invention.

The notion of utilizing an in-line, continuous process for producing laminates has been previously suggested as a possibility. See, for instance, PCT publication WO 96/19346 to common assignee. Like airplane models drawn prior to the Wright brothers' successful flight, however, actually breathing life into the notion of creating the in-line, continuous process of the present invention has only come about through the long-term, concentrated efforts of highly qualified individuals. Furthermore, the laminate produced by the process of the present invention exhibits unexpected properties as will be discussed in more detail below.

### **Summary of the Invention**

The present invention is directed to an in-line, continuous process for preparing a laminate of film and a nonwoven web wherein the process includes the steps of: forming an unaged film, simultaneously forming an unaged nonwoven web, and laminating said oriented unaged film and said unaged nonwoven web to form a

laminate within 1-60 seconds of forming said oriented, unaged film and said unaged nonwoven web.

### **Brief Description of the Drawings**

Figure 1 is a perspective view of a process for the in-line, continuous forming of a laminate according to the present invention from an unaged film and unaged nonwoven web.

Figures 2a, b and c represent a schematic side view of a three-step process of the prior art for forming a laminate from an aged film and aged nonwoven web.

Figure 3 is a schematic side view of a two-step process of the prior art for forming a laminate from an aged film and unaged nonwoven web.

Figure 4 is a partially cut-away top plan view of an exemplary personal care absorbent article, in this case a diaper, which may utilize the film/nonwoven laminate according to the present invention.

Figure 5 is a perspective view of the threading means of the present invention.

Figure 6a is a schematic side view of the prior art Machine Direction Orienter.

Figure 6b is a schematic side view of the Machine Direction Orienter of the present invention.

### **Detailed Description of the Invention**

The present invention is directed to an in-line, continuous process for making a laminate of film and nonwoven web wherein the film and nonwoven web are made simultaneously, and are therefore nascent or unaged when directly formed into a laminate. Forming such a laminate through an in-line, continuous process has heretofore been unknown. In the process of the present invention, the film is formed in-line while the nonwoven web is formed, the newly formed film and nonwoven web being immediately laminated upon forming. Hence, the laminate is formed from "unaged" film and "unaged" nonwoven web. As used herein, the term "unaged," which is synonymous with "green," means that the film and nonwoven web are immediately laminated upon forming, for example, neither the film nor the nonwoven web is pre-wound into a roll prior to forming into a laminate.

As used herein the term "nonwoven fabric or web" means a web having a structure of individual fibers or threads which are interlaid, but not in an identifiable manner as in a knitted fabric. Nonwoven fabrics or webs have been formed from many



processes such as for example, meltblowing processes and spunbonding processes. The basis weight of nonwoven fabrics is usually expressed in ounces of material per square yard (osy) or grams per square meter (gsm) and the fiber diameters useful are usually expressed in microns. (Note that to convert from osy to gsm, multiply osy by 33.91).

Referring to Figure 1, the unaged film 10 of the present invention may be made from polymers which are capable of being formed into films and then bonded to an unaged nonwoven web 30. As used herein, the term "polymer" generally includes but is not limited to, homopolymers, copolymers, such as for example, block, graft, random and alternating copolymers, terpolymers, etc. and blends and modifications thereof. Furthermore, unless otherwise specifically limited, the term "polymer" shall include all possible geometrical configurations of the molecule. These configurations include, but are not limited to isotactic, syndiotactic and random symmetries. Such polymers include but are not limited to extrudable thermoplastic polymers such as polyolefin or a blend of polyolefins. More particularly, useful polyolefins include polypropylene and polyethylene. Other useful polymers include those described in U.S. Patent No. 4,777,073 to Sheth, assigned to Exxon Chemical Patents Inc., such as a copolymer of polypropylene and low density polyethylene or linear low density polyethylene. Additional polymers useful in the present invention include flexible polyolefins. As used herein the term "flexible polyolefin" refers to polyolefin materials containing propylene based polymer with controlled regions of atactic polypropylene units to achieve a desired crystallinity such as described in co-assigned U.S. Patent No. 5,910,136 to Hetzler and Jacobs; the entire contents of which are incorporated herein by reference in its entirety. Further description of such flexible polyolefins can be found in U.S. Patent No. 5,723,546 to Sustic and assigned to Rexene Corporation.

Other useful polymers for the unaged film of the present invention include elastomeric thermoplastic polymers. Such polymers include those made from block copolymers such as polyurethanes, copolyether esters, polyamide, polyether block copolymers, ethylene vinyl acetates (EVA), block copolymers having the general formula A-B-A' or A-B like copoly(styrene/ethylene-butylene), styrene-poly(ethylene-propylene)-styrene, styrene-poly(ethylene-butylene)-styrene, polystyrene poly(ethylene-butylene) polystyrene, poly(styrene/ethylene-butylene/ styrene) and the like. Specifically, the elastomeric thermoplastic polymers include: polyester elastomeric materials such as, for example, those available under the trade designation HYTREL® from E. I. du Pont de Nemours and Company; polyester block amide copolymers such as, for example, available in various grades under the trade

designation PEBAX® from ELF Atochem Inc. of Glen Rock, New Jersey; and polyurethane elastomeric materials such as, for example, those available under the trademark ESTANE® from B. F. Goodrich & Co. or MORTHANE® from Morton Thiokol Corporation.

Elastomeric polymers have been used in the past for many applications but are somewhat limited by their intrinsic properties. These materials have recently been joined by a new class of polymers which has excellent barrier, breathability and elasticity. The new class of polymers is referred to as single site catalyzed polymers such as "metallocene" polymers produced according to a metallocene process.

Such metallocene polymers are available from Exxon Chemical Company of Baytown, Texas under the trade name EXXPOL® for polypropylene based polymers and EXACT® for polyethylene based polymers. Dow Chemical Company of Midland, Michigan has polymers commercially available under the name ENGAGE®. Preferably, the metallocene polymers are selected from copolymers of ethylene and 1-butene, copolymers of ethylene and 1-hexene, copolymers of ethylene and 1-octene and combinations thereof. For a more detailed description of the metallocene polymers and the process for producing same which are useful in the present invention see coassigned PCT Application WO 98/29246 to Gwaltney et al., which is incorporated herein by reference in its entirety.

The unaged film 10 may be a multi-layered film which may include a core layer, or "B" layer, and one or more skin layers, or "A" layers on either side of the core layer. Any of the polymers discussed above are suitable for use as a core layer of a multi-layered film. Any of the fillers disclosed herein are suitable for use in any film layer.

The skin layer will typically include extrudable thermoplastic polymers and/or additives which provide specialized properties to the film 10. Thus, the skin layer may be made from polymers which provide such properties as antimicrobial activity, water vapor transmission, adhesion and/or antiblocking properties. The polymers are thus chosen for the particular attributes desired. Examples of possible polymers that may be used alone or in combination include homopolymers, copolymers and blends of polyolefins as well as ethylene vinyl acetate (EVA), ethylene ethyl acrylate (EEA), ethylene acrylic acid (EAA), ethylene methyl acrylate (EMA), ethylene butyl acrylate (EBA), polyester (PET), nylon (PA), ethylene vinyl alcohol (EVOH), polystyrene (PS), polyurethane (PU), and olefinic thermoplastic elastomers which are multistep reactor products wherein an amorphous ethylene propylene random copolymer is molecularly dispersed in a predominately semicrystalline high polypropylene monomer/low ethylene monomer continuous matrix.

Suitable polymers for the "A" layer are available commercially under the trade designation "Catalloy" from the Himont Chemical Company of Wilmington, Delaware. Specific commercial examples are Catalloy, KS 357P, KS-084P and KS-057P. Other suitable polymers include polymers which are semi-crystalline/amorphous or heterophasic in character. Such polymers are disclosed in European Patent Application EP 044467.1 A3 (based on Application number 91103014.6), European Patent Application EP 0472946 A2 (based on Application number 91112955.9), European Patent Application EP 0400333 A2 (based on Application number 90108051.5), U.S. Patent number 5,302,454 and U.S. Patent number 5,368,927.

For a more detailed description of films having core and skin layers see PCT WO 96/19346 to McCormack et al. assigned to common assignee which is incorporated herein by reference in its entirety.

The films can be made from breathable or non-breathable materials. In addition the films can be apertured. In-forming the films, the films may be coextruded to increase bonding and alleviate die lip build-up. The films may be filled with such fillers as micropore developing fillers, e.g. calcium carbonate; opacifying agents, e.g. titanium dioxide; and antiblock additives, e.g. diatomaceous earth.

Fillers may be incorporated for developing micropores during orientation of the film resulting in breathable films. Some films are made breathable by adding filler particles to the film during the film forming process. Once the particle-filled film has been formed, it is then either stretched or crushed to create pathways through the film. Generally, to qualify as being "breathable" for the present invention, the resultant laminate should have a water vapor transmission rate (WVTR) of at least about 250 g/m<sup>2</sup>/24 hours as may be measured by a test method as described below.

As used herein, a "micropore developing filler" is meant to include particulates and other forms of materials which can be added to the polymer and which will not chemically interfere with or adversely affect the extruded film but are able to be uniformly dispersed throughout the film. Generally, the micropore developing fillers will be in particulate form and usually will have somewhat of a spherical shape with average particle sizes in the range of about 0.5 to about 8 microns. As used herein, a "micron" means a micrometer. The film will usually contain at least about 30 percent of micropore developing filler based upon the total weight of the film layer. A unique advantage to the present invention is that less micropore developing filler may be required than has been previously used. Both organic and inorganic micropore developing fillers are contemplated to be within the scope of the present invention provided that they do not interfere with the film formation process, the

breathability of the resultant film or its ability to bond to another layer such as a fibrous polyolefin nonwoven web.

Examples of micropore developing fillers include calcium carbonate ( $\text{CaCO}_3$ ), various kinds of clay, silica ( $\text{SiO}_2$ ), alumina, barium sulfate, sodium carbonate, talc, magnesium sulfate, titanium dioxide, zeolites, aluminum sulfate, cellulose-type powders, diatomaceous earth, magnesium sulfate, magnesium carbonate, barium carbonate, kaolin, mica, carbon, calcium oxide, magnesium oxide, aluminum hydroxide, pulp powder, wood powder, cellulose derivative, polymer particles, chitin and chitin derivatives. The micropore developing filler particles may optionally be coated with a fatty acid, such as stearic acid, or a larger chain fatty acid than starch such as behenic acid, which may facilitate the free flow of the particles (in bulk) and their ease of dispersion into the polymer matrix. Silica-containing fillers may also be present in an effective amount to provide antiblocking properties.

The unaged film 10 can be made from either cast or blown film equipment, can be coextruded and can be embossed if so desired. Preferably, the film will be made by cast extrusion. There are two known types of cast extrusion equipment: chill cast and cast embossing. Typical cast embossed extrusion is shown in Figure 2a wherein the film is extruded into a pair of cast embossing rollers 42. Chill cast extrusion is the preferred film extrusion technique of the present invention for reasons as discussed below. Turning to Figure 1, in chill cast extrusion, the polymer enters coextrusion film apparatus 40 and is extruded through core film extruder 47 and skin film extruder 45. The polymer is coextruded into die head 43 and then only one roll, a chill cast roll 49, is used to receive the extruded film 10, not two rolls as is done in cast embossed extrusion. The advantage of chill cast over cast embossed extrusion is that chill cast extrusion is more efficient when extruding wide sheets of film and manufacturing speeds can be increased. Also, cast embossed extrusion imparts a texture to the film versus the smooth surface of the chill cast extruded film. In other words, films made from chill cast extrusion may be cast from a 160 inch (406 cm) die, resulting in an approximately 154 inch (391 cm) wide film, produced at speeds as much as 400 fpm (122 m/min) and even 500 fpm (150 m/min). Films made by cast embossed extrusion, on the other hand, frequently only result in 140-145 inch (356 -368 cm) wide film at speeds of only 300 fpm (91.5 m/min), since the distance traveled by the extruded material from the extruder to the rolls is typically greater than that of chill cast extruded material. Films made by chill cast extrusion, therefore, exhibit less necking-in, (width reduction), and have a more uniform gauge profile.

Additionally, the film 10 can be stretched or oriented by passing the film through a film stretching unit 20 as shown in Figure 1. The stretching reduces the film gauge or thickness from an initial gauge of 1.5-2.0 mils to an effective final gauge of 0.5 mils (12.7 microns) or less. Generally, this stretching may take place in the cross-machine direction or in the machine direction. As used herein, the term "machine direction" or MD means the length of a fabric in the direction in which it is produced. The term "cross machine direction" or CD means the width of fabric, i.e. a direction generally perpendicular to the MD.

The unaged nonwoven web 30 as illustrated in Figure 1, in a laminate 32 containing the unaged film 10 of the present invention, may be formed from a number of processes including, but not limited to, spunbonding and meltblowing processes. Such nonwoven webs can be necked polypropylene spunbond, crimped polypropylene spunbond, elastomeric spunbond or meltblown fabrics produced from elastomeric resins. As used herein, the term "necked" refers to constricting in at least one dimension by processes such as, for example, drawing or gathering.

Suitable fibers for forming the unaged nonwoven web 30 include natural and synthetic fibers as well as bicomponent and multi-component/polymer fibers. A plurality of nonwoven web layers in laminate 32 may also be used according to the present invention. Examples of such materials can include, for example, spunbond/meltblown composites and spunbond/meltblown/spunbond composites such as are taught in Brock et al., U.S. Patent No. 4,041,203 which is incorporated herein by reference in its entirety.

As used herein, the term "spunbonded fibers" refers to small diameter fibers which are formed by extruding through one or more extruders, which is attached to one or more banks made up of at least transfer piping and spinplates, to produce molten thermoplastic material as filaments from a plurality of fine, usually circular, capillaries in a spinneret with the diameter of the extruded filaments then being rapidly reduced as by, for example, in Appel et al., U.S. Patent No. 4,340,563; Matsuki, et al., U.S. Patent No. 3,802,817; Dorschner et al., U.S. Patent No. 3,692,618; Kinney, U.S. Patent Nos. 3,338,992 and 3,341,394; Hartman, U.S. Patent No. 3,502,763; and Dobo et al., U.S. Patent No. 3,542,615. Spunbond fibers are generally not tacky when they are deposited onto a collecting surface. Spunbond fibers are generally continuous and have average diameters larger than 7 microns, more frequently, between about 10 and 20 microns.

As used herein the term "meltblown fibers" means fibers formed by extruding a molten thermoplastic material through a plurality of fine, usually circular, die capillaries

as molten threads or filaments into converging high velocity, usually hot, gas (e.g. air) streams which attenuate the filaments of molten thermoplastic material to reduce their diameter, which may be to microfiber diameter. Thereafter, the meltblown fibers are carried by the high velocity gas stream and are deposited on a collecting surface to form a web of randomly dispersed meltblown fibers. Such a process is disclosed, for example, in U.S. Patent No. 3,849,241 to Butin et al. Meltblown fibers are microfibers which may be continuous or discontinuous, are generally smaller than 10 microns in average diameter, and are generally tacky when deposited onto a collecting surface and may not require a separate bonding step.

As used herein the term "microfibers" means small diameter fibers having an average diameter not greater than about 75 microns, for example, having an average diameter of from about 0.5 microns to about 50 microns, or more particularly, microfibers may often have an average diameter of from about 2 microns to about 40 microns. Another frequently used expression of fiber diameter is denier, which is defined as grams per 9000 meters of a fiber and may be calculated as fiber diameter in microns squared, multiplied by the density in grams/cc, multiplied by 0.00707. A lower denier indicates a finer fiber and a higher denier indicates a thicker or heavier fiber. For example, the diameter of a polypropylene fiber given as 15 microns may be converted to denier by squaring, multiplying the result by 0.89 g/cc and multiplying by 0.00707. Thus, a 15 micron polypropylene fiber has a denier of about 1.42 ( $15^2 \times 0.89 \times 0.00707 = 1.415$ ). Outside the United States the unit of measurement is more commonly the "tex", which is defined as the grams per kilometer of fiber. Tex may be calculated as denier/9.

Many polyolefins are available for fiber production according to the present invention, for example fiber forming polypropylenes include Exxon Chemical Company's Escorene® PD 3445 polypropylene and Himont Chemical Company's PF-304. Polyethylenes such as Dow Chemical's ASPUN® 6811A linear low density polyethylene, 2553 LLDPE and 25355 and 12350 high density polyethylene are also suitable polymers. The polyethylenes have melt flow rates, respectively, of about 26, 40, 25 and 12. Many other polyolefins are commercially available.

Processes for forming films and processes for forming fibrous nonwoven webs are generally known. Likewise, forming a laminate of film/nonwoven web via two- and three-step processes are also known. As illustrated in Figure 2, a three-step prior art process generally includes forming the aged film 10' in continuous which includes winding the resulting film onto a roll 14. The film 10' may then be placed in storage, usually for as much as 2 days to 4 weeks. The aged nonwoven web 30' is formed in

another step which likewise is wound onto a roll 16. Each of the rolls 14 and 16 of aged film 10' and aged nonwoven web 30' respectively is then unwound, the film 10' may be stretched or oriented in film stretching unit 44, and laminated in a third step to form laminate 32'.

A two-step prior art process generally includes pre-forming either the film or nonwoven web which is wound into a roll, then unwinding the pre-formed material in-line of a process which is simultaneously forming the opposite material. For example and as shown in Figure 3, the aged film 10' is pre-formed and wound into a roll 14. The unaged nonwoven web 30 is formed while unwinding the pre-rolled, aged film 10' to form the laminate 32".

Forming a film/nonwoven laminate through an in-line, continuous process in practice has heretofore been unknown. As mentioned above, Reliability Theory would dictate that very low yields would be expected from the process of the present invention, which would tend to lead one of ordinary skill in the art away from pursuing the present invention. In practice, overall process efficiency for a typical film making process is usually on the order of 74% (90% raw material yield and 82% machine utilization), whereas overall process efficiency for a typical spunbond (nonwoven web) process is usually on the order of 89% (97% raw material yield and 92% machine utilization). By combining these two processes into the in-line, continuous process of the present invention, one would expect an overall process efficiency of approximately 66% with no particular product advantages anticipated. The process efficiency of the present invention has in fact been found to be on the order of at least 70% and even at least 75% in addition to the property improvements exhibited by products made according to the present invention. At high production rates, such an increase in efficiency is very meaningful, and is a function of the process conditions and parameters as described herein.

In the process of the present invention, the unaged film 10 is formed in-line while the unaged nonwoven web 30 is formed, which is immediately laminated upon forming. Hence, the laminate 32 is formed from "unaged" film and "unaged" nonwoven web. By "immediately" what is meant is that the unaged film 10 and unaged nonwoven web 30 are joined together into a laminate 32 without first winding either layers. Preferably, the layers will be laminated within 1 to 60 seconds, more preferably 1 to 30 seconds, often even in the range of 1 to 10 seconds, from the time each layer is made. It is through this immediate lamination, brought about by the ability to simultaneously process the unaged film 10 and unaged nonwoven web 30, that the unexpected and

surprising improvements in peel strength and hydrohead are found as will be highlighted in more detail below.

More specifically, Figure 1 illustrates a general approach to forming and orienting an unaged film 10 such as one according to the present invention. Referring to Figure 1, the unaged film 10 is formed from a coextrusion film apparatus 40 such as a cast or blown unit as was previously described above. Typically the apparatus 40 will include one or more polymer extruders such as core film extruder 47 and skin film extruder 45. The unaged film 10 is extruded onto a chill cast roll 49.

From the coextrusion film apparatus 40, the unaged film 10 is directed via a threading means 18 (as shown in Figure 5 and discussed in more detail below) through a film stretching unit such as a machine direction orienter (MDO) 20 (as shown more particularly in Figure 6b). Such an apparatus 20 has a plurality of stretching rollers 46 which progressively stretch and thin the film 10 in the machine direction of the film which is in the direction of travel of unaged film 10 through the process. After exiting the MDO 20, the unaged film 10 should have a maximum thickness of approximately 12 microns (0.47 mils).

Machine direction stretching of the prior art, shown generally at 44 in Figure 6a, generally included use of 6 stretching rollers 46' which generally utilized 3-4 heating zones and 1 or more cooling zones. In other words, some stretching rollers would be heated and some would be cooled. The prior art process also included nip rolls 21 positioned to maintain contact of the film with the stretching rollers 46 while stretching under conditions of conductive heating by the roller. It is believed that stretching while heating against the rollers of the prior art results in uneven heating, over-necking and a poor CD property profile. Figure 6b, on the other hand, illustrates the wind-up of film through the MDO 20 according to an aspect of the present invention. The amount and type of stretching is a function of positioning of the nip rolls 21 against the stretching rollers 46 which may be a chrome-plated roll. The nip rolls 21 are crowned rolls which provide uniform nipping effect against the stretching rollers 46. The nip rolls 21 of the present invention are positioned such that wrap of the unaged film 10 is maximized against the stretching rollers 46, for example, 270° around the diameter of the roll. In accordance with one embodiment of the invention, the MDO 20 stretches the film after it leaves the nip rolls 21 which means that stretching occurs in the space between the rollers, thereby avoiding the problems of the prior art. The MDO is configured with at least seven stretching rollers. Each stretching roller may be heated from ambient to 250°F (121°C) or cooled from ambient to 55°F (13°C) and is driven separately. The rate at which each



stretching roller rotates is in the range of 380 - 1550 fpm (116 - 473 m/min). Therefore, the final draw ratio may be in the range of 1.00 to 4.08. This MDO as described is designed to maximize flexibility and is more versatile than the film stretching apparatus of the prior art in that it allows for processing and stretching of many different types of films under many different process conditions. It has been found in the unaged film of the present invention that the film stress whitens at a lower stretch ratio, meaning that not as much stretching is required. There are several advantages to this phenomenon. One such advantage is that less stretching of the film means that the film will not be as stressed as the higher stretch ratios used in the prior art which ultimately results in less film breaks and decreased film defects. The net effect is that the process may be run at higher rates, in addition to producing higher yields. Another advantage, as alluded to hereinbefore, is that less micropore developing filler will be required, meaning that more polymer will be utilized in the composition. Such compositions will result in tougher films meaning that thinner films may be utilized in the final laminate and resulting articles.

The threading means 18 provide for feeding or threading the formed unaged film through the entire continuous process, releasing the film once the film is laminated to the nonwoven web. A typical threading means is illustrated in Figure 5 wherein three ropes are used in concert to grasp one end of the film sheet and feed it through the process. The three ropes function essentially as three fingers in that one rope is in proximal contact with one side of the sheet while the other two ropes are in proximal contact with the opposite side of the sheet and the three ropes compress together to hold the sheet end and feed it through the process.

As the unaged film is forming at one point of the machine, a nonwoven web is simultaneously being formed. Referring again to Figure 1, a conventional nonwoven web forming apparatus 48, such as a spunbond machine, is used to form unaged nonwoven web 30. The long, essentially continuous fibers 50 are deposited onto a forming wire 52 as an unbonded web 54 and the unbonded web 54 is then sent through a pair of bonding rolls 56 to bond the fibers together. One or both of the rolls are often heated to aid in bonding. The temperature at which the bonding rolls 56 are heated is in the range of 250 - 350°F (121 - 177°C). Typically, one of the rolls 56 is also patterned so as to impart a discrete bond pattern with a prescribed bond surface area to the unaged nonwoven web 30. This is known as thermal point bonding as described more fully below. The other roll is usually a smooth anvil roll but this roll may also be patterned if so desired. Once unaged film 10 has been sufficiently stretched and the unaged nonwoven web 30 has been formed, the two layers are immediately,

as described above, brought together and laminated to one another in this continuous process using a pair of laminating rolls or other means 58.

In a preferred mode, the smooth anvil roll is positioned to the side of the unaged nonwoven web 30 to which the unaged film 10 will be attached. In other words, the smooth side of unaged nonwoven web 30 will be attached to the unaged film 10, which results in better bonding of the two layers together.

When the unaged film 10 and unaged nonwoven web 30 are bonded through the use of heat and/or pressure, laminating means 58 such as laminating rolls may be used. As with the bonding rolls 56, the laminating rolls 58 may be heated and thermal point bonding may be used. The temperature at which the laminating rolls are heated is in the range of 200 - 275°F (93 - 135°C). At least one of the rolls may be patterned to create a discrete bond pattern with a prescribed bond surface area for the resultant laminate 32. Generally, the maximum bond point surface area for a given area of surface on one side of the laminate 32 will not exceed about 50 percent of the total surface area. There are a number of discrete bond patterns which may be used. See, for example, Brock et al., U.S. Patent No. 4,041,203.

"Thermal point bonding" involves passing a fabric or nonwoven web of fibers to be bonded between a heated calender roll and an anvil roll. The calender roll is patterned in some way so that the entire nonwoven web is not bonded across its entire surface. Many patterns for calender rolls have been developed for functional as well as aesthetic reasons. As will be understood by those skilled in the art, bond area percentages are, of necessity, described in approximations or ranges since bond pins are normally tapered and wear down over time. As those skilled in the art will also recognize, references to "pins/in.<sup>2</sup>" and "bonds/in.<sup>2</sup>" are somewhat interchangeable since the anvil pins will create bonds in the substrate in essentially the same sizes and surface relationship as the pins on the anvil. One example of a pattern has points and is the Hansen Pennings or "H&P" pattern with about 200 bonds/square inch as taught in U.S. Patent No. 3,855,046 to Hansen and Pennings. The H&P pattern has square point or pin bonding areas wherein each pin may have a side dimension of 0.038 inches (0.965 mm), for example, resulting in a pattern having a bonded area of about 30%. Another typical point bonding pattern is the expanded Hansen and Pennings or "EHP" bond pattern which produces a bond area of about 15% to 18% which may have a square pin having a side dimension of 0.037 inches (0.94 mm), for example, and a pin density of about 100 pins/in<sup>2</sup>. Another typical point bonding pattern designated "714" has square pin bonding areas wherein each pin may have a side dimension of 0.023 inches, for example, for a bond area of 15% to 20% and about 270 pins/in<sup>2</sup>.

Other common patterns include a "Ramisch" diamond pattern with repeating diamonds having a bond area of 8% to 14% and 52 pins/in.<sup>2</sup>, a HHD pattern, which comprises point bonds having about 460 pins/in.<sup>2</sup> for a bond area of about 15% to about 23%, as well as a wire weave pattern looking as the name suggests, e.g. like a window screen and having a bond area of 15% to 20% and 302 bonds/in.<sup>2</sup>. Another bond pattern for a spunbond facing web is a "S" weave pattern as described in coassigned US patent application Serial No. 929,808 filed on 15-September-1997 in the names of McCormack, Fuqua, and Smith, and entitled "Nonwoven Bonding Patterns Producing Fabrics with Improved Strength and Abrasion Resistance" which is incorporated herein by reference in its entirety. Typically, the percent bonding area varies widely from around 10% to around 30% of the area of the nonwoven web.

Once the laminate 32 exits the laminating rolls 58, it may be wound up into a roll 60 for subsequent processing. Alternatively, the laminate 32 may continue in-line for further processing or conversion.

As will be explained in more detail below, a surprising and unexpected improvement of the present invention lies in its increase in peel strength and hydrohead which results in an advantage in converting the laminate into an article such as a personal care absorbent article. One major disadvantage of presently known laminates is the tendency to delaminate prior to or during conversion. Such delamination results in various problems with commercial production as well as increased waste-products. As one of ordinary skill in the art is aware, an increase in either peel strength or hydrohead usually results in a decrease in the other property. An advantage of the present invention lies in that both peel strength and hydrohead are increased at the same time.

Additionally, the process shown in Figure 1 may be used to create a laminate of more than two layers. The previously described process can be modified to feed a supply 16 of a second aged nonwoven web 30' into the laminating rolls 58 on a side of film 10 which is opposite that of the other unaged nonwoven web 30. Also contemplated by the present invention is the forming of a second unaged nonwoven web directly in-line of the continuous process as described above for unaged nonwoven web 30. Such three layer laminates are particularly useful in medical and industrial protective garment applications. Similarly, other aged or unaged film layers may be combined.

As has been stated previously, the laminate 32 may be used in a wide variety of applications, not the least of which includes as a component of personal care absorbent articles such as diapers, training pants, incontinence devices and feminine

hygiene products such as sanitary napkins. An exemplary article 80, in this case a diaper, is shown in Figure 4. Referring to Figure 4, most such personal care absorbent articles 80 include a liquid permeable top sheet or liner 82, a back sheet or outercover 84 and an absorbent core 86 disposed between and contained by the top sheet 82 and back sheet 84. Articles 80, such as diapers, may also include some type of fastening means 88 such as adhesive fastening tapes or mechanical hook and loop type fasteners to maintain the garment in place on the wearer.

The laminate 32 may be used to form various portions of the article including, but not limited to the top sheet 82 and the back sheet 84. If the laminate is to be used as the top sheet 82, it will most likely be apertured or otherwise made to be liquid permeable. When using the laminate as back sheet 84, it is usually advantageous to place the nonwoven side facing out away from the user. In addition, in such embodiments it may be possible to utilize the nonwoven portion of the laminate as the loop portion of the hook and loop combination of fastening means 88.

Other uses for the film/nonwoven web laminates according to the present invention include, but are not limited to, surgical drapes and gowns, wipers, barrier materials and articles of clothing or portions thereof including such items as workwear and lab coats.

The advantages and other characteristics of the present invention are best illustrated by the following examples:

### **Examples**

Samples of the present invention were prepared as described below. The samples were then subjected to the following tests:

**Peel test:** In peel or delamination testing a laminate was tested for the amount of tensile force required to pull the film layer apart from the nonwoven web layer. Values for peel strength were obtained using a width of fabric sample of approximately 4 inch (CD) x 6 inch (MD) (102 x 152 mm), held between 1 x 4 inch (25 x 102 mm) long parallel clamps (or jaws) and extending at a constant rate of extension of  $12 \pm 0.4$  in./min. ( $300 \pm 10$  mm/min.). Masking tape or some other suitable material was applied to the film side of the sample to prevent the film from ripping apart during the test. The masking tape was on only one side of the laminate and so did not contribute to the peel strength of the sample. The sample was delaminated by hand a sufficient amount to allow it to be clamped into position usually approximately 2 inches (51 mm). The sample was clamped, for example, in an Instron Model TM, available from the Instron

Corporation, 2500 Washington St., Canton, MA 02021, or a Sintech tensile tester available from Sintech, Inc., P.O. Box 14226, Research Triangle Park, North Carolina 27709-4226. The sample was then pulled apart for a distance of 2 in. (51 mm) at 180° of separation and the average peel strength was recorded in grams.

**Tensile Test:** The tensile test measured breaking strength and elongation or strain of a fabric when subjected to unidirectional stress. The results were expressed in grams to break and percent stretch before breakage. Higher numbers indicate a stronger, more stretchable fabric. The term "peak load" means the maximum load or force, expressed in units of weight, required to break or rupture the sample in a tensile test. The term "energy" means the total energy under a peak load versus elongation curve as expressed in weight-length units. The term "strain" or "percent stretch" means the increase in length of a sample during a tensile test expressed as a percentage. Values for peak load, energy, and strain were obtained using a fabric sample of 3 x 6 in. (76 x 152 mm), a 3 in. (76 mm) clamp width, a gauge length of 3 in. (76 mm), and a constant rate of extension of 12 inches/min. (300 mm/min.), where the entire sample width was gripped in the clamps. The specimen was clamped, for example, in an 1130 Instron, available from the Instron Corporation, or a Thwing-Albert Model INTELLECT II available from the Thwing-Albert Instrument Co., 10960 Dutton Rd., Philadelphia, Pennsylvania 19154.

**Ball Burst:** This test measures the bursting strength of textile fabrics that exhibit a high degree of ultimate elongation and was conducted in accordance with ASTM D 3787-89. The bursting strength is defined as the force or pressure required to rupture a textile by distending it with a force, applied at right angles to the plane of the fabric, under specified conditions. The pressure was generated by forcing a polished steel ball against the sample until the sample burst using an Instron tensile tester modified for the ball burst test. The pressure was then recorded for that bursting strength in lbf (N). The sample was conditioned to moisture equilibrium for testing textiles in the standard atmosphere as prescribed in ASTM Practice D 1776.

**Breathability Test:** A measure of the breathability of a fabric is the water vapor transmission rate (WVTR), which for sample materials are calculated in accordance with ASTM Standard E96-80. Circular samples measuring three inches (7.6 cm) in diameter are cut from each of the test materials. A CELGARD® 2500 sheet from Celanese Separation Products of Charlotte, NC is used as a control. CELGARD® 2500 sheet is a microporous polypropylene sheet. Three samples are prepared for each material. The test dish is a number 60-1 Vapometer pan distributed by Thwing-Albert Instrument Company of Philadelphia, PA. One hundred

milliliters of water are poured into each Vapometer pan and individual samples of the test materials and control material are placed across the open tops of the individual pans. Screw-on flanges are tightened to form a seal along the edges of the pan, leaving the associated test material or control material exposed to the ambient atmosphere over a 6.5 centimeter diameter circle having an exposed area of approximately 33.17 square centimeters. The pans are placed in a forced air oven at 100°F (32°C) for 1 hour to equilibrate. The oven is a constant temperature oven with external air circulating through it to prevent water vapor accumulation inside. A suitable forced air oven is, for example, a Blue M Power-O-Matic 60 oven distributed by Blue M Electric Company of Blue Island, Illinois. Upon completion of the equilibration, the pans are removed from the oven, weighed and immediately returned to the oven. After 24 hours, the pans are removed from the oven and weighed again. The preliminary test water vapor transmission rate values are calculated as follows:

Test WVTR = (grams weight loss over 24 hours) x 315.5 g/m<sup>2</sup>/24 hours.

The relative humidity within the oven is not specifically controlled.

Under predetermined set conditions of 100°F (32°C) and ambient relative humidity, the WVTR for the CELGARD® 2500 control has been defined to be 5000 grams per square meter for 24 hours. Accordingly, the control sample is run with each test and the preliminary test values are corrected to set conditions using the following equation:

$$\text{WVTR} = (\text{Test WVTR} / \text{control WVTR}) \times (5000 \text{ g/m}^2 / 24 \text{ hours})$$

**Hydrohead:** A measure of the liquid barrier properties of a fabric is the hydrohead test. The hydrohead test determined the height of water (in centimeters) which the fabric would support before a predetermined amount of liquid passed through. A fabric with a higher hydrohead reading indicated it had a greater barrier to liquid penetration than a fabric with a lower hydrohead. The hydrohead test was performed according to Federal Test Standard 191A, Method 5514 using a Textest FX-3000 Hydrostatic Head Tester available from Marlo Industries, Inc., P.O. Box 1071, Concord, North Carolina.

**Standard Deviation:** Standard deviation as used in these examples represents a measure of dispersion and measures the average distance between a single observation and its mean. This is useful for understanding how variable a set of data may be. For example, the standard deviation may be used to allow one to predict failure rates and/or to determine how much variability is acceptable in a final product. For the Examples as described below, forty-four samples of material were tested for

each of the properties. Twenty-two of the samples were taken from the edge of the laminate sheet as it came off of the production line and twenty-two were taken from the center of the laminate. This number of samples represents a statistically significant sample size.

The formula used for standard deviation was:

$$\sqrt{\frac{\sum(x_i - \bar{x})^2}{n - 1}}$$

In the formula, "n" is the number of observations. The use of n-1 in the denominator instead of the more natural n was used because if n (instead of n-1) were used, a biased estimate of the population standard deviation would result. The use of n-1 corrected for this bias with small sample sizes. The distance from each observation ( $x_i$ ) to the calculated average ( $\bar{x}$ ) provides the basis for measuring variability. The closer these observations are to the average, the smaller the standard deviation. If all observations are the same, the standard deviation would be zero. The deviations are squared due to the average being the "fulcrum" of the data (a balance point between those observations greater than the average and those less than the average). If these deviations were not squared, the sum would be zero. The square root of the sum is then taken to get the value back into the units of the original data.

Statistics are utilized herein to compare the Examples as noted below. The following is an explanation of the statistical analysis that was performed for these Examples. One formal method of making statistical inferences from samples to a population is through hypothesis testing. The sampling distributions of statistics provides a method to substantiate or refute a hypothesis about group means. The t-test is the most commonly used method to evaluate the differences in means between two normally distributed groups. There are two t-tests available. The standard t-test for independent samples is based on the assumption that the variances in the two groups are the same (homogeneous) and a pooled standard deviation is used. If the variances in the two groups are widely different then the t-test with separate variance estimates is used. The equality of variances assumption can be verified with the F test. The F test is used to compare the variances of two normal populations by looking at the ratio of the sample variances. The null hypothesis is testing that  $\text{variance1} = \text{variance2}$  (or  $\text{variance1}/\text{variance2} = 1$ ).

In the fundamental two-sample test, the statistical hypothesis is that the two means are equal ( $\text{Var1}=\text{Var2}$ ). The alternative hypothesis is that a difference exists ( $\text{Var1}-\text{Var2} <> 0$ ). If the probability level is less than your chosen alpha level (0.05), you reject the null hypothesis of equal means and conclude that the means are different. The p-level reported with a t-test represents the probability of error involved in accepting our research hypothesis about the existence of a difference.

### **Example 1**

A laminate was prepared from an unaged film and unaged nonwoven web according to the present invention. The unaged film was coextruded as a three layer structure, otherwise known as an A/B/A film.

The "B" layer, or core layer, was made up of 44.5 wt.% linear low density polyethylene (LLDPE) available under the trade designation 3310 as manufactured by the Dow Chemical Company ("Dow"); 5.3 wt.% low density polyethylene (LDPE) available under the trade designation 4012 as manufactured by Dow; 50 wt.% behenic acid coated calcium carbonate available under the trade designation FilmLink® 2029 as manufactured by ECC International, Inc. of Sylacauga, Alabama; and 2000 ppm antioxidant available under the trade designation B900 as manufactured by Ciba Specialties Company of Tarrytown, New York.

The "A" layers, otherwise known as skin or outer layers, on opposite sides of the core layer, were made up of 50.4 wt.% ethylene vinyl acetate under the trade designation 768.36 as manufactured by the Exxon Chemical Company of Houston, Texas; 49.1 percent Catalloy under the trade designation KS 357P as manufactured by Montell USA Incorporated of Wilmington, Delaware; and 5000 ppm anti-oxidant available under the trade designation B900 as manufactured by the Ciba Specialties Company.

The three layer film was chill cast extruded as described above and at conditions as described below. The exiting melt temperature for the skin layers was measured to be approximately 365°F (185°C) and for the core was approximately 420°F (215°C). The skin layers comprised approximately 2.5 wt.% of the total film composition.

### **FILM PROCESSING CONDITIONS**

Thruput	Core	Skin	BW prior to stretching
Lb/hr	rpm	rpm	Osy
3800	31.9	30.3	176

BW means Basis Weight



The film was passed through a Machine Direction Orienter (MDO) having 7 rolls under the conditions as tabulated below. Final film draw was defined as the ratio of the speed of the last roll to the first roll. The film was stretched 3.5X and the resulting stretched film had a basis weight after stretching of 0.54 osy. By saying the film was stretched 3X it is meant that, for example, a 1 meter length of film would be stretched to a resultant length of 3 meters.

#### MDO PROCESS CONDITIONS

	Speed	Draw	Draw	TEMPS
	fpm (M/min)	%	Ratio	°F (°C)
Roll #1	410 (125)	98.0%	1.02	200 (93.3)
Roll #2	419 (128)	76.0%	1.32	200 (93.3)
Roll #3	551 (168)	98.0%	1.02	195 (90.6)
Roll #4	562 (171.3)	38.0%	2.63	190 (87.8)
Roll #5	1479 (450.8)	101.0%	0.99	70 (21.1)
Roll #6	1464 (446.2)	102.0%	0.98	70 (21.1)
Roll #7	1436 (437.7)	103.0%	0.97	210 (99)

The unaged nonwoven web was prepared from a spunbonding process as described above using two nonwoven web forming apparatus as shown in Figure 1. The nonwoven web was formed from a polypropylene having a melt flow rate (MFR) of 38 available under the trade designation E5D47 as manufactured by Union Carbide Corporation of Danbury, Connecticut and 2 wt. % titanium dioxide concentrate available from Standridge Color Corporation of Social Circle, Georgia. The fibers were extruded through two extruders and two banks, then drawn down to an average diameter of 15-20 microns and deposited onto the forming wire. The speed of the forming wire was adjusted so as to result in a nonwoven web having a basis weight of 0.5 oz/sq yd (osy) (17 g/m<sup>2</sup>), which was then thermally point bonded through wire-weave patterned bonding rolls heated to 315°F (157°C).

The unaged nonwoven web was then conveyed beneath the MDO to be laminated to the stretched unaged film using an anvil and a C-star patterned laminating rolls under conditions of heat and pressure, with lamination conditions of 353 PLI (61,800 N/M), the top anvil laminating rolls at a temperature of 220°F (104.4°C), and the bottom pattern laminating rolls at a temperature of 280°F (137.8°C).

The stretched unaged film was immediately laminated to the unaged nonwoven web within less than 5 seconds of being made. The laminate was passed through the laminating rolls in such a fashion that the spunbond layer was adjacent the pattern roll and the film layer was adjacent the smooth anvil roll. A statistically significant sample group was tested and the properties shown in Tables 1a and 1b. Unless otherwise

noted, the basis weight in the following numbered tables represents the basis weight of the laminate.

### **Example 2**

A laminate was prepared from an unaged film and unaged nonwoven web as described above for Example 1, with the following different processing conditions.

#### **MDO PROCESS CONDITIONS**

	Speed	Draw	Draw	TEMPS
	fpm (M/min)	(%)	Ratio	F (°C)
Roll #1	444 (135)	98.0%	1.02	210 (99)
Roll #2	453 (138)	56.0%	1.32	200 (93.3)
Roll #3	809 (247)	65.0%	1.02	200 (93.3)
Roll #4	1245 (379)	85.0%	2.63	200 (93.3)
Roll #5	1464 (446)	101.0%	0.99	70 (21.1)
Roll #6	1450 (442)	101.0%	0.98	70 (21.1)
Roll #7	1435 (438)	102.0%	0.97	220 (104.4)

Final film draw ratio was 3.23.

#### **Lamination Conditions**

Laminating Rolls	Pressure	Temperature
	PLI (N/m <sup>2</sup> )	°F (°C)
Top (Anvil)	353 (61,800)	214 (101.1)
Bottom (Pattern)	353 (61,800)	269 (131.7)

A statistically significant sample group was tested and the properties shown in Tables 2a and 2b.

### **Comparative Example**

A laminate was prepared from an aged film and an unaged nonwoven web. The aged film was coextruded as an A/B/A film as described below and manufactured by the Huntsman Packaging Company of Salt Lake City, Utah.

The "B" layer was made up of the same composition as in Example 1. The "A" layers were made up essentially the same as in Example 1 except that 45.1 wt.% Catalloy was employed along with 4 wt.% Superfloss® diatomaceous earth as available from Celite Corporation of Lompoc, California. The skin layer comprised approximately 3.3 wt.% of the total film composition.

The aged film was allowed to age for 4 days in storage.

The aged film was then oriented in a conventional machine direction orienter manufactured by the Marshall and Williams Company, under the following conditions:

**MDO PROCESSING CONDITIONS**

	Speed	Draw	Draw	TEMPS
	fpm (M/min)	%	Ratio	°F (°C)
Unwind	375 (114)	99.21%	1	
Preheat roll 1	378 (115)	76.99%	1.3	225 (107.2)
Preheat roll 2	491 (150)	98.00%	1.02	200 (93.3)
Slow draw roll	501 (153)	28.98%	3.45	200 (93.3)
Fast draw roll	1729 (527)	102.01%	0.98	77 (25)
Anneal roll 1	1695 (517)	101.92%	0.98	77 (25)
Anneal roll 2	1663 (507)	107.64%	0.93	210 (99)

The final film draw ratio was 4.44x.

A spunbond unaged nonwoven web which was made up of the same composition as in Example 1 was then laminated to the aged film under the following conditions:

**Lamination Conditions**

Laminating Rolls	Pressure	Temperature
	PLI (N/m <sup>2</sup> )	°F (°C)
Top (Anvil)	360 (63,000)	216 (102.2)
Bottom (Pattern) Film	360 (63,000)	236 (113.3)

A statistically significant sample group was tested and the properties shown in Tables 3a and 3b.

**Conclusions:**

Tables were created summarizing statistical analysis of these above property tables as can be seen below in Tables 4a and 4b.

The data shows unexpected improved barrier performance and laminate strength in both Examples of the invention for both hydrohead and peel strength, respectively, as compared to the two-step process (Comparative Example). Contrary to popular belief, which prescribes that there is usually a compromise of one of these properties (hydrohead and peel) if an increase is found in the other property, the process of the present invention produces materials having improvements in both properties simultaneously. This is extremely desirable in articles produced from the products of this invention.

Additionally, the process further produced materials which were strong enough for use in products as described herein which has been demonstrated by sufficient

tensile strength and ball burst properties. Having thus described the invention in detail, it should be apparent that various modifications can be made in the present invention without departing from the spirit and scope of the following claims.

We claim:

1. An in-line, continuous process for preparing a laminate, comprising the steps of:
  - a) forming a film, while;
  - b) forming a nonwoven web, thereafter;
  - c) immediately joining said film and said nonwoven web to form a laminate.
2. The process of claim 1 wherein said forming of said film comprises cast film extrusion or blown film extrusion.
3. The process of claim 1 wherein said forming of said film comprises chill cast extrusion.
4. The process of claim 1 wherein said forming of said film results in a maximum film thickness of less than about 12 microns.
5. The process of claim 1 wherein said forming of said nonwoven web comprises formation of one or more types of fiber selected from the group comprising spunbond and meltblown fibers.
6. The process of claim 1 wherein said forming of said nonwoven web comprises bonding the fibers of said nonwoven web.
7. The process of claim 1 wherein said joining of said film and said nonwoven web occurs within 1-60 seconds of said forming of said film and said forming of said nonwoven web.
8. The process of claim 1 wherein said joining of said film and said nonwoven web comprises thermal bonding.
9. The process of claim 1 wherein the overall process efficiency is at least about 70%.
10. The process of claim 1 wherein said laminate has greater peel strength and greater hydrohead values than similar laminates constructed of aged materials.

11. An in-line, continuous process for preparing a laminate, comprising the steps of:
  - a) forming a film, then;
  - b) immediately stretching said film to render it breathable, while;
  - c) forming a nonwoven web, thereafter;
  - d) immediately joining said breathable film and said nonwoven web to form a laminate.
12. The process of claim 11 wherein said forming of said breathable film comprises cast film extrusion or blown film extrusion.
13. The process of claim 11 wherein said forming of said breathable film comprises chill cast extrusion.
14. The process of claim 11 wherein said stretching of said film comprises drawing said film by greater than 1.00 and up to about 4.08 times its original length.
15. The process of claim 14 wherein the magnitude of said stretching of said film required to make said film breathable is reduced compared to the magnitude of stretching required to make similar aged film breathable.
16. The process of claim 15 wherein said reduced magnitude of said stretching of said film required to make said film results in higher rates and yields during said stretching of said film than during the stretching required to make similar aged film breathable.
17. The process of claim 11 wherein said stretching of said film results in a maximum film thickness of less than about 12 microns.
18. The process of claim 11 wherein said forming of said nonwoven web comprises formation of one or more types of fiber selected from the group comprising spunbond and meltblown fibers.
19. The process of claim 11 wherein said forming of said nonwoven web comprises bonding the fibers of said nonwoven web.

20. The process of claim 11 wherein said joining of said breathable film and said nonwoven web occurs within 1-60 seconds of said forming of said breathable film and said simultaneous forming of said nonwoven web.
21. The process of claim 11 wherein said joining of said breathable film and said nonwoven web comprises thermal bonding.
22. The process of claim 11 wherein the overall process efficiency is at least about 70%.
23. The process of claim 11 wherein said laminate has greater peel strength and greater hydrohead values than similar laminates constructed of aged materials.
24. A laminate prepared by an in-line, continuous process, the steps of which comprise:
  - a) forming a film, while;
  - b) forming a nonwoven web, thereafter;
  - c) immediately joining said film and said nonwoven web to form a laminate.
25. The laminate of claim 23 wherein said film is breathable.
26. The laminate of claim 23 wherein said nonwoven web comprises one or more types of fiber selected from the group comprising spunbond and meltblown fibers.
27. The laminate of claim 23 wherein said film and said nonwoven web comprise polyolefins.
28. The laminate of claim 23 wherein said film and said nonwoven web are thermally bonded together.
29. The laminate of claim 23 wherein said laminate has greater peel strength and greater hydrohead values than similar laminates constructed of aged materials.

30. An absorbent article comprising a liquid-permeable liner, a liquid-impermeable outercover, and an absorbent core disposed therebetween, wherein at least one of the liquid-impermeable outercover and the liquid permeable liner comprises a laminate according to claim 23.



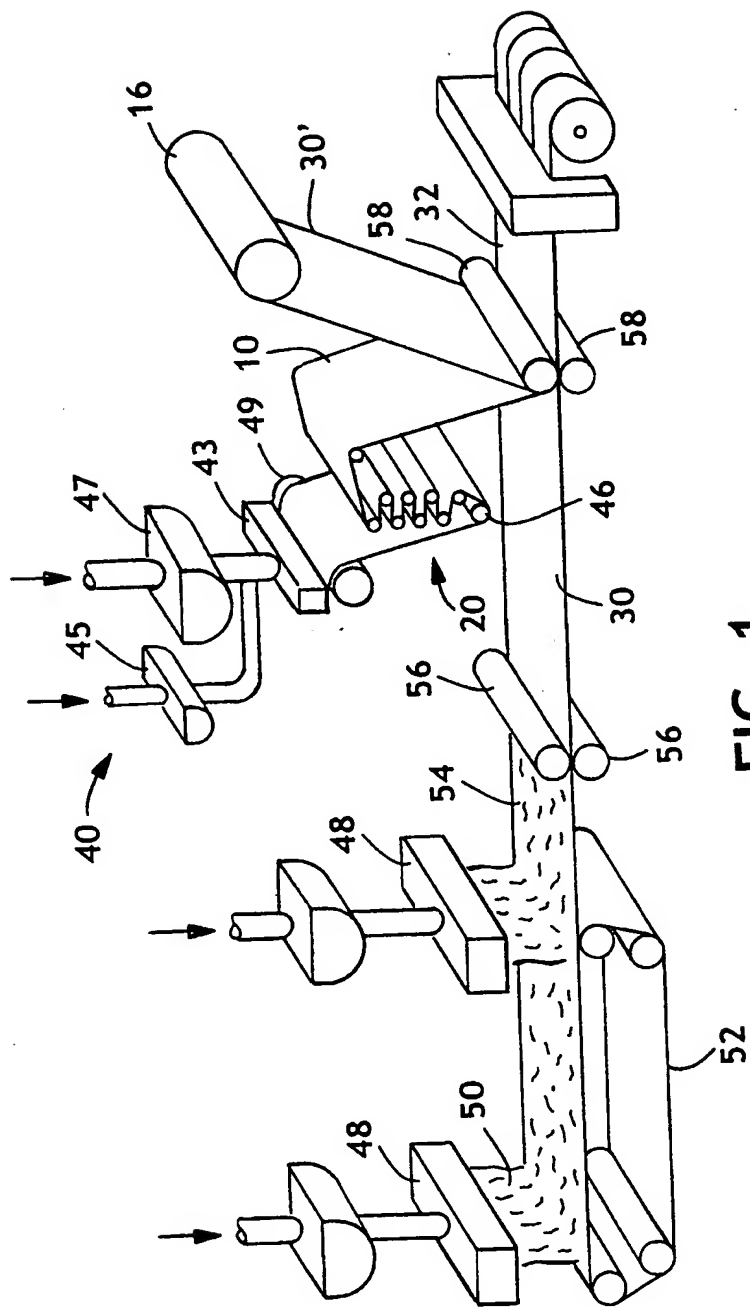


FIG. 1

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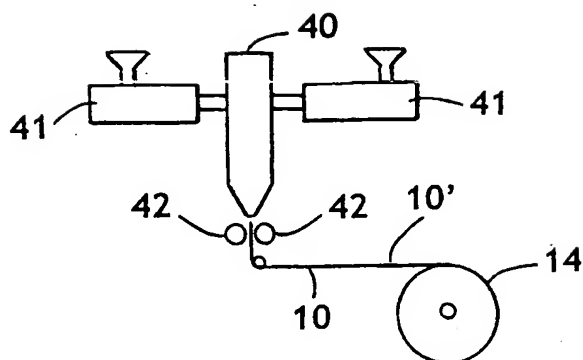


FIG. 2A

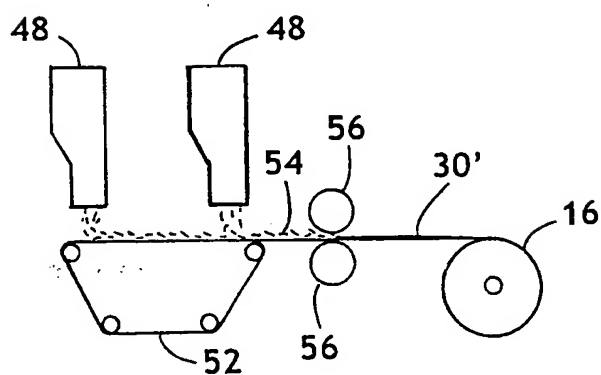


FIG. 2B

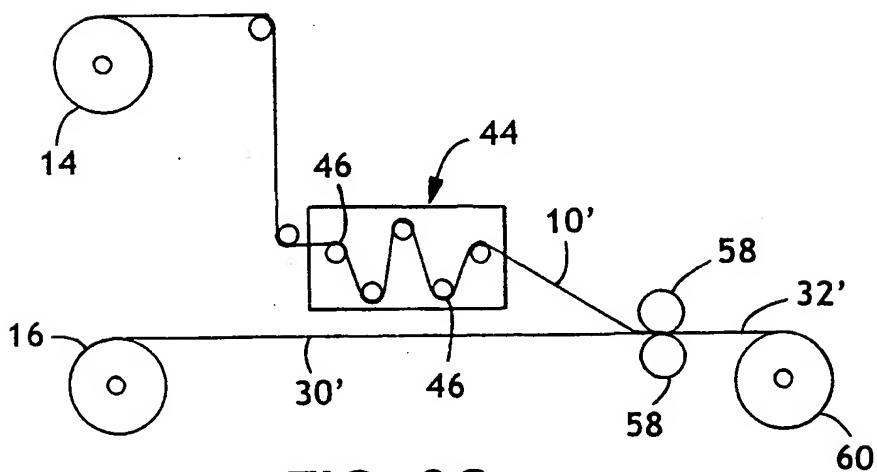


FIG. 2C

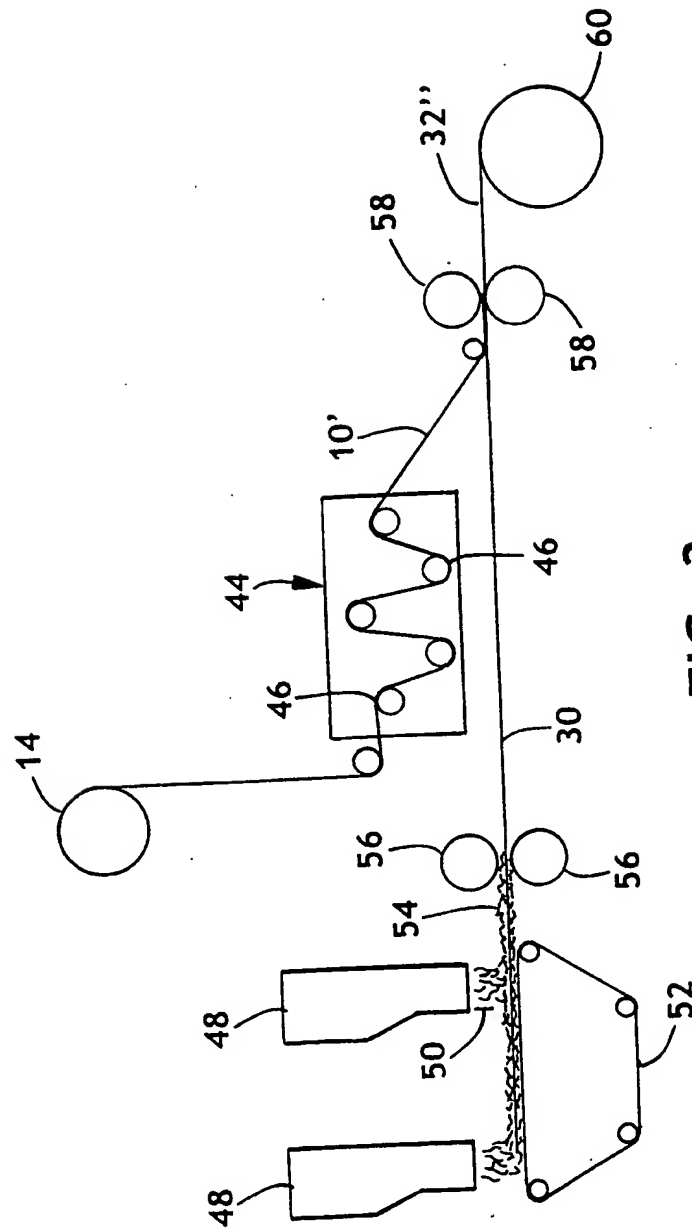


FIG. 3

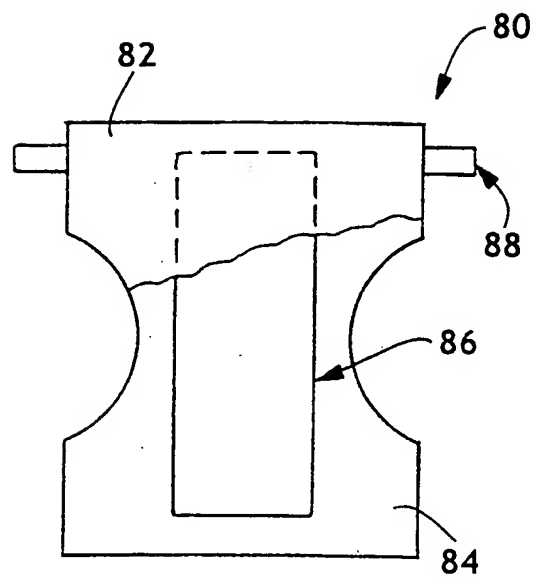
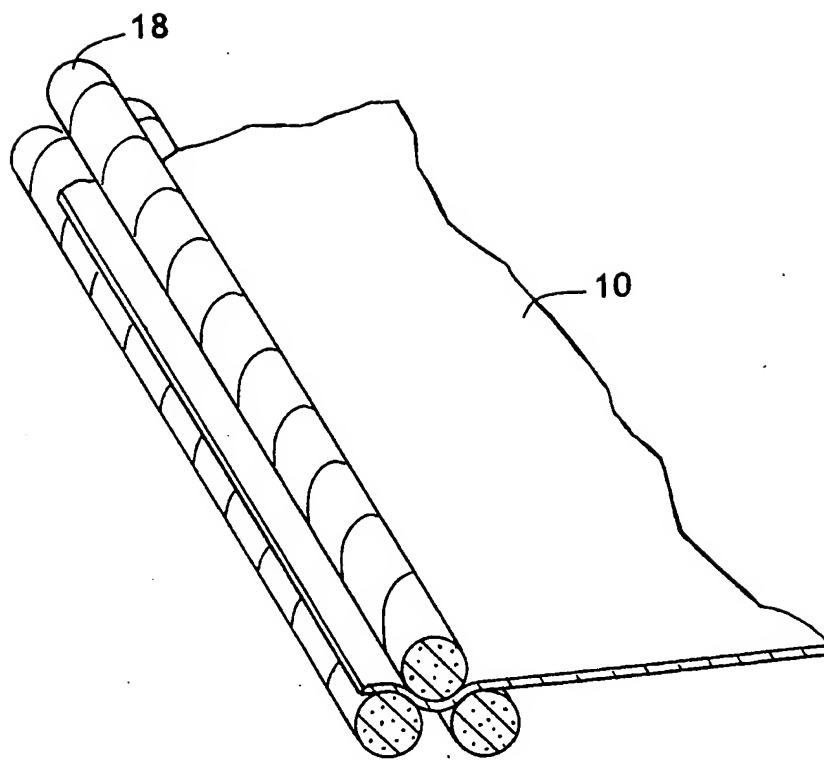


FIG. 4



**FIG. 5**

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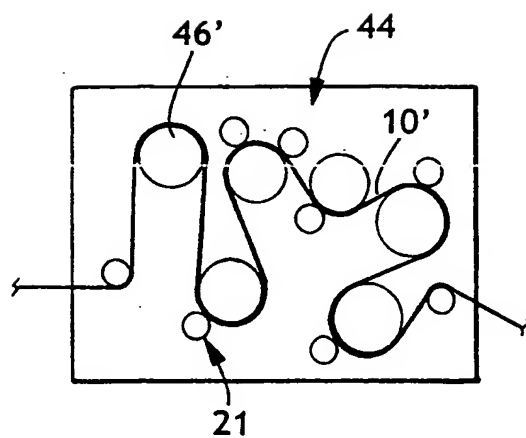


FIG. 6A

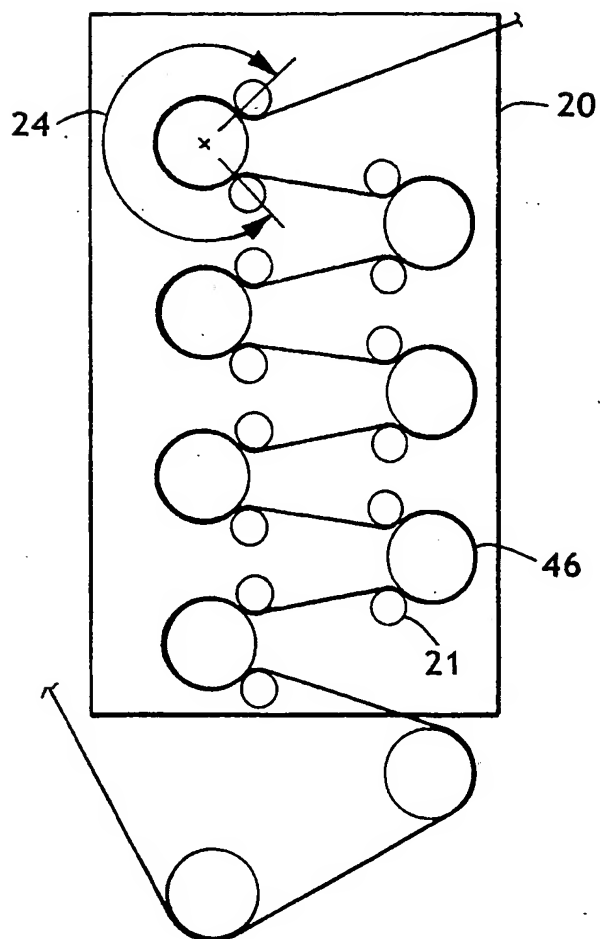


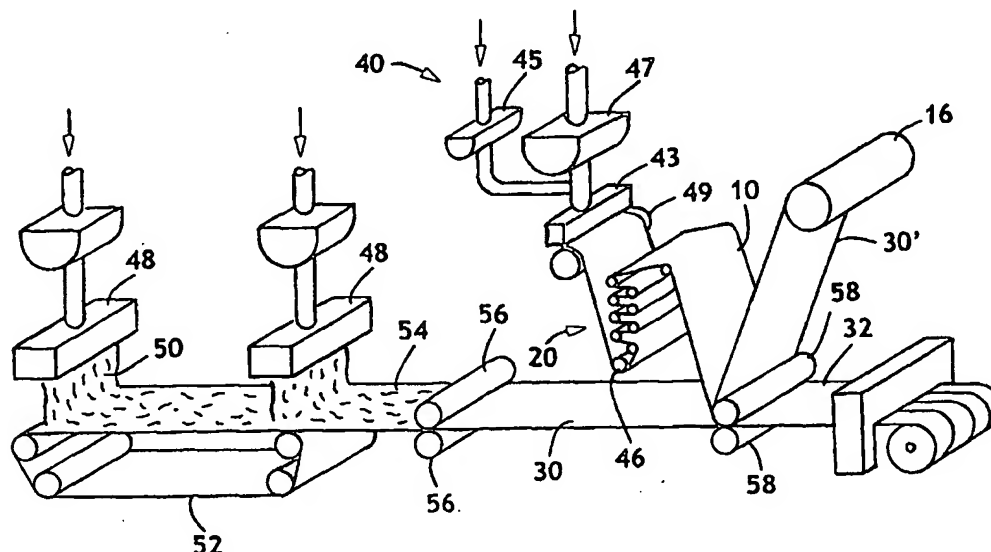
FIG. 6B



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>7</sup> : <b>D04H 13/00, B32B 5/26, 27/08, B29C 47/02, B32B 27/12, A61F 13/15</b>		A3	(11) International Publication Number: <b>WO 00/16974</b>
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(74) Agents: MOYLES, Lisa, J. et al.; 401 N. Lake St., Neenah, WI 54956 (US).			

(54) Title: PROCESS FOR MAKING A LAMINATE OF UNAGED FILM AND AN UNAGED NONWOVEN WEB AND PRODUCTS PRODUCED THEREFROM



## (57) Abstract

The present invention is directed to an in-line, continuous process for making a laminate of film and nonwoven web wherein the film and nonwoven web are made simultaneously, and are therefore nascent or unaged when directly formed into a laminate. In the process of the present invention, the film is formed in-line while the nonwoven web is formed, the newly formed film and nonwoven web being immediately laminated upon forming. Hence, the laminate is formed from "unaged" film and "unaged" nonwoven web. As used herein, the term "unaged", which is synonymous with "green", means that the film and nonwoven web are immediately laminated upon forming, for example, neither the film nor the nonwoven web is pre-wound into a roll prior to forming into a laminate.

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## INTERNATIONAL SEARCH REPORT

Int'l. Application No.

PCT/US 99/21500

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 D04H13/00 B32B5/26 B32B27/08 B29C47/02 B32B27/12  
A61F13/15

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## C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	DE 43 11 867 A (COROVIN GMBH) 13 October 1994 (1994-10-13) column 5, line 12 - line 14; figures column 5, line 59 - column 6, line 3 column 6, line 42 - line 45; claims 22,23 column 6, line 46 - line 54	1,2,6,8, 24-28
X	WO 96 19346 A (KIMBERLY CLARK CO) 27 June 1996 (1996-06-27) cited in the application  page 12, line 1 - page 13, line 9; figure 3 page 10, line 10 - page 11, line 4	1,2,5,6, 8,11,12, 18,19, 21, 24-28,30
X	EP 0 691 203 A (KIMBERLY CLARK CO) 10 January 1996 (1996-01-10) figure 3	1-30
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A	PATENT ABSTRACTS OF JAPAN vol. 015, no. 053 (M-1079), 7 February 1991 (1991-02-07) & JP 02 286313 A (TOSOH CORP), 26 November 1990 (1990-11-26) abstract	1-30
A	EP 0 395 381 A (TOSOH CORP) 31 October 1990 (1990-10-31)	1-30
A	DATABASE WPI Section Ch, Week 198816 Derwent Publications Ltd., London, GB; Class A23, AN 1988-109857 XP002132564 & JP 63 059531 A (KAO CORP), 15 March 1988 (1988-03-15) abstract	1-30
P,X	WO 98 58799 A (KIMBERLY CLARK CO) 30 December 1998 (1998-12-30) figure 3	1-30

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 99/21500

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE 4311867 A	13-10-1994	WO 9423947 A	27-10-1994
WO 9619346 A	27-06-1996	AU 698621 B	05-11-1998
		AU 4603596 A	10-07-1996
		BR 9510475 A	26-05-1998
		CA 2208535 A	27-06-1996
		CN 1175226 A	04-03-1998
		EP 0799128 A	08-10-1997
		FR 2728786 A	05-07-1996
		GB 2296216 A, B	26-06-1996
		PL 320886 A	10-11-1997
		TR 960585 A	21-07-1996
		ZA 9510604 A	03-07-1996
EP 0691203 A	10-01-1996	AU 2046595 A	14-12-1995
		BR 9502680 A	28-05-1996
		CA 2148392 A	07-12-1995
		FR 2720684 A	08-12-1995
		GB 2290052 A	13-12-1995
		JP 7329249 A	19-12-1995
		ZA 9504516 A	13-05-1996
JP 02286313 A	26-11-1990	JP 2748541 B	06-05-1998
		EP 0395381 A	31-10-1990
		US 5089075 A	18-02-1992
EP 0395381 A	31-10-1990	JP 2286313 A	26-11-1990
		JP 2748541 B	06-05-1998
		JP 2762586 B	04-06-1998
		JP 3049922 A	04-03-1991
		JP 2824915 B	18-11-1998
		JP 3070748 A	26-03-1991
		US 5089075 A	18-02-1992
JP 63059531 A	15-03-1988	JP 1898518 C	23-01-1995
		JP 4067499 B	28-10-1992
WO 9858799 A	30-12-1998	AU 8059798 A	04-01-1999

